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CERAMIC MATRIX COMPOSITES IN SIMULATED SSME ENVIRONMENTS

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ABSTRACT

Future Space Shuttle Main Engine (SSME)-type rocket engines can benefit from the use of fiber-reinforced ceramic matrix composites (FRCMC). Ceramics reinforced with long continuous fibers exhibit improved tolerance to severe thermomechanical and environmental exposures. An in-house NASA-Lewis program to evaluate the durability of FRCMC in simulated SSME environments is described. Primary tests involve multiple (one second) exposures of FRCMC specimens in a hydrogen/oxygen rocket test rig. This rig generates surface heating rates of 1000 to 2500°C/second. FRCMC durability evaluation involves measurement of retained strength as a function of thermal shock severity and number of upshock cycles. Preliminary test results for monolithic silicon nitride (Si_3N_4) and silicon carbide (SiC), and one type of silicon based FRCMC, are presented. The test data are examined in terms of simple thermal shock theory.

INTRODUCTION

Reusable rocket engines for future earth-to-orbit space missions must operate longer, withstand more duty cycles, and be more efficient than present generation engines [1]. Today the most advanced reusable rocket engine is the space shuttle main engine (SSME). Superalloy turbopump blades, stator vanes and other hot gas flow path components of this hydrogen/oxygen burning engine have limited durability. For improved efficiency, the next generation Space Transport Booster Engine (STBE) and Space Transport Main Engine (STME) will require materials with even greater temperature capability and durability.

Several materials have the potential to significantly outperform the superalloys now used in advanced rocket engines. Candidates include ceramics, intermetallics, and carbon/carbon composites. These materials have a lower density and can operate at higher temperatures than superalloys (Figure 1). Of the candidate materials for use above 2000°F , ceramics exhibit the best tolerance to the aggressive gaseous environment of a rocket engine. Complex structural components have been fabricated out of monolithic silicon nitride and silicon carbide. Extensive tests show these components have promise for long term use in automotive turbine engines at temperatures approaching 2500°F (1370°C) [2].

Carpenter [1] has shown that properly designed monolithic ceramics also have potential as turbopump elements for use in the hostile environment of an advanced SSME-type engine. He notes, however, that the thermal transients are far more severe in SSME applications than in an automotive turbine engine. The thermal stresses associated with these transients are detrimental to monolithic ceramics which have low toughness. Because of their low toughness, the load carrying capability of monolithic ceramics may be reduced by flaws introduced during processing and service. This results in a low but finite probability of brittle (catastrophic) failure of monolithic ceramics under thermal shock conditions. In addition, catastrophic material failure may lead to other engine components being severely damaged.

Reinforcing ceramics with long continuous ceramic fibers offers the potential for significant improvement in reliability and durability. In particular, fiber reinforced ceramic matrix composites (FRCMC) exhibit toughnesses almost an order of magnitude greater than monolithic ceramics. The high toughness of FRCMC results in their strength being essentially insensitive to many processing and service-induced flaws that would severely degrade monolithic ceramics. Flaws may originate during service by environmental attack, particle impact, or local stresses generated by thermal shock. High FRCMC toughness thus may imply a

longer use life in engine applications than monolithic ceramics. Also, because of the long fibers, failure in FRCMC occurs in a noncatastrophic or graceful manner. This is particularly important for potential use in man-rated and high-mission-value unmanned engine applications.

The advantages of FRCMC have only recently been demonstrated. Thus, their development is less mature than that of monolithic ceramics. As such, there is a limited technology base. And no extensive experience exists for detailed design, fabrication and testing of actual or simulated turbine hardware.

Recognizing the high potential for FRCMC, NASA Lewis initiated a project to assess the feasibility of using them for components in advanced rocket engines. The project consists of an in-house research program and a two phase, 60 month contract. These combined efforts will lead to a technology plan to implement the introduction of FRCMC into future generation earth-to-orbit rocket engine turbine components. The work breakdown and scheduling for the contract effort and parallel in-house property screening program are shown in Figure 2. Phase I of the contract will identify the benefits and assess the potential for using FRCMC in advanced earth-to-orbit rocket engine turbine components. Phase II includes tasks to design, fabricate and test full scale prototype components. Testing of components will be in a simulated rocket engine environment.

The initial in-house efforts at NASA Lewis focus on the thermal shock resistance of FRCMC. This is a critical parameter in the incorporation of a ceramic material in the hot section of a rocket engine. Rapid heating (thermal upshock) test data will be generated for a variety of advanced FRCMC materials. Residual strength data, in combination with fracture analysis and nondestructive evaluation studies, will provide information needed to assess durability. This in turn will be used to develop methods for material improvement. Parallel tests will be conducted on monolithic ceramics to demonstrate and evaluate the advantages of FRCMC. This paper describes the in-house NASA-Lewis thermal shock test program. Preliminary test results for SiC and Si_3N_4 monolithic ceramics and one type of FRCMC are presented.

IN-HOUSE EXPERIMENTAL PROGRAM

The thermal downshock (rapid cooling) of monolithic ceramic materials has been studied extensively. A number of parameters exist to predict performance (see appendix). In contrast, little information exists in the literature on the thermal upshock of monolithic ceramics, much less FRCMC. This disparity is the result of a number of factors. Primary among these is the ease of

conducting a thermal downshock test. Also, surface tensile forces created during rapid cooldown represent the most severe condition for many applications.

Brindley and Nesbitt [3] have described the thermal shock rig which is being used in this study. It is basically a small hydrogen/oxygen rocket engine. The test specimens are positioned in the exit of the engine in the rocket exhaust. Figure 3 is an approximate curve for the thermal shock transient generated at the ceramic/metal interface in a ceramic-coated (60 mils thick) metallic substrate [3]. Also shown is the approximate gas temperature gradient. This latter temperature gradient roughly corresponds to the temperature at the surface of the sample. Thus the approximate upshock rate on the sample surface is greater than 1900°C/second. The oxygen/hydrogen mass flow ratio (O/F ratio) for this test was 1:4. Altering the O/F ratio allows control of the surface upshock rate from 1000 to 2500 °C/second.

The FRCMC sample configuration is a rectangular bar 12.7 mm wide by approximately 3 mm thick by 76 mm long. Figure 4 is a drawing of the test specimen holder. The holder orients the specimen at various angles to the rocket engine exhaust. Varying the specimen orientation simulates a variety of turbine blade and vane angles.

The test matrix (Table I) involves 12 tests (3 samples/test) of each material type. Post exposure characterization shall include measurement of room temperature flexural strength and fractography. All specimens will be nondestructively characterized before and after thermal shock. There are a number of applicable nondestructive tests. One promising test is the measurement of internal friction. A schematic of the NASA Lewis internal friction apparatus is shown in Figure 5. Preliminary studies on monolithic and composite ceramics indicate this technique is sensitive to the microstructural damage that may occur as a result of thermal shock. These measurements will be correlated with the strength and fractography data.

The initial materials for evaluation include monolithic SiC and Si_3N_4, and two types of SiC/SiC# having a laminated 2-D woven fiber architecture. The monolithic ceramics will be used to establish test procedures and serve as a baseline for demonstrating the advantages of FRCMC in thermal shock conditions. Other composite materials considered for testing include: SiC/RBSN, SiC/Si_3N_4, oxide/SiC, oxide/Si_3N_4, and carbon/SiC. Availability and the existence of a baseline property database

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will dictate material selection. Most of the available FRCMC are in the early stages of development and SiC is the usual matrix. However, downshock test results on monolithic ceramics indicate that Si_3N_4-based materials have better thermal shock resistance than SiC-based materials [4].

The results of preliminary thermal upshock testing are presented in Table II. The monolithic SiC specimens failed in less than three thermal shock cycles with a surface heating rate approximately 1200°C per second (O/F ratio of 1:0). The Si_3N_4 monolithic materials performed better than the SiC. This is in agreement with thermal downshock literature and theory. However, failure did occur after a relatively low number of cycles. A SiC/SiC composite (Nicalon* fibers in a chemical vapor infiltrated (CVI) SiC matrix)+ was also tested. In contrast to the monolithic ceramics, the composite survived 5 upshock cycles at a ΔT of ~1500°C (O/F ratio 1:2). The CVI coating sustained minor damage, but visually there was no apparent loss of structural integrity. These encouraging results support the hypothesis that FRCMC will outperform monolithic ceramics in the severe thermal shock conditions of rocket turbine engines.

Fiber arrangement or architecture can play an important role in the thermal shock durability of FRCMC. Initial tests will concentrate on laminated composites with a rectangular geometry. As testing progresses, alternate fiber architectures and specimen geometries will be studied. The Structures Division of NASA-Lewis will help define a fiber architecture having improved thermal upshock resistance. Future tests will evaluate a FRCMC rocket engine turbopump vane or blade with optimum fiber architecture.

SUMMARY

Fiber reinforced ceramic matrix composites (FRCMC) have a number of attributes that make them attractive for use in rocket engine turbopump applications. An in-house test program in support of a component oriented contract effort has been developed at NASA Lewis. FRCMC durability will be assessed under simulated SSME environmental conditions. Preliminary thermal upshock results and mechanistic analysis show FRCMC have greater potential than monolithic ceramics to survive the severe thermal upshock environment of present and future generation rocket engine turbopumps.

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Appendix

Thermal Shock of Ceramics

The durability of monolithic ceramics in thermal downshock conditions is often evaluated by employing two parameters. The first relates to the onset of crack initiation and the second relates to crack propagation resistance. Equations (1) and (2) show the general form of the crack initiation R and crack propagation R' parameters, respectively [5].

Crack Initiation

$$R = \frac{\sigma_f(1-\nu)}{\alpha\ A\ E} \tag{1}$$

Crack Propagation

$$R' = \frac{E\ \gamma}{\sigma_f^2\ (1-\nu)} \tag{2}$$

where:

σ_f = fracture stress
ν = Poisson's ratio
α = coefficient of thermal expansion
A = thermal conductivity and geometry constant
E = elastic modulus
γ = work of fracture

Equations (1) and (2) show a nearly inverse relationship between R and R'. Thus, for most monolithic materials, a high thermal shock fracture initiation resistance and a high thermal shock propagation resistance cannot be achieved simultaneously. In dealing with monolithic ceramic materials, crack initiation is the more important factor. This is because the onset of crack initiation is often sufficient to create flaws of a critical size. This in turn results in catastrophic material failure. The crack initiation parameter predicts that high values of elastic modulus (E) and coefficient of thermal expansion (α) result in a lower crack initiation and shock resistance.

Two silicon-based ceramic materials that have received the most attention for potential use in severe thermal environments are SiC and Si_3N_4. Since SiC has higher values for E and α than Si_3N_4, it is not surprising that Si_3N_4 has a better thermal downshock resistance than SiC [1]. Similar behavior was observed in preliminary thermal upshock testing of monolithic SiC and Si_3N_4 specimens in the NASA-LeRC thermal upshock rig (Table II).

The key to the superior behavior of FRCMC specimens lies in the properties of the constituents. First, the thermal shock resistance of the matrix benefits from the crack propagation parameter, R'. After a matrix crack is initiated, failure does not occur immediately if insufficient energy is available for the crack to propagate across the fibers. This is a result of a large increase in the work of fracture term, γ. The measured work of fracture of a SiC/SiC composite has been determined to be nearly two orders of magnitude greater than monolithic SiC [6]. The resistance to crack propagation increases accordingly.

Second, small diameter fibers have much greater crack initiation resistance to thermal shock than large monolithic specimens. This relates to the geometric portion of the constant A in equation (1). As specimen size decreases, the geometric factor decreases at an increasing rate. At extremely small dimensions,the other terms in equation (1) reduce to second order effects. Becher et al [7] have demonstrated that very small ceramic test specimens are, for all practical purposes, thermal shock resistant. Figure 6 shows the critical temperature difference (ΔT) for thermal shock damage as a function of specimen thickness [7]. Nicalon SiC fibers have an inverse thickness value near $1x10^5$ cm^{-1}. If this value is applied to Figure 6, an increase of several orders of magnitude in the predicted critical ΔT over monolithic ceramics is predicted.

The combined effect of small diameter fibers resisting thermal shock crack initiation and the ability of the fiber/matrix interactions to resist matrix crack propagation explains the minimal thermal shock degradation expected for FRCMC. This is supported by preliminary results for a SiC/SiC composite (Table II).

Table I. Thermal Shock Test Matrix

Condition	Number of Specimens		
	ΔT^* = 1200 C	1400 C	1600 C
One Shock Cycle	3	3	3
Ten Shock Cycles	3	3	3
Twenty Shock Cycles	3	3	3
Cycle to Failure	3	3	3

*ΔT = Approximate surface temperature change in a one second pulse.

Table II. Preliminary Thermal Upshock Test Results

Material	Results
SiC Monolithic	• 3 samples tested at ΔT = 1200 C (O/F 1.0). All failed in less than 3 cycles.
Si_3N_4 Monolithic	• 2 samples tested at ΔT = 1200 C (O/F 1.0). No failures in 5 cycles. • 3 samples tested at ΔT = 1500 C (O/F 1.2). All failed in 5 or less cycles.
SiC/SiC Composite	• 1 sample tested 5 cycles at ΔT = 1200 C (O/F 1.0) No significant damage. • 1 sample tested at ΔT = 1500 C (O/F 1.2). Progressive minor damage during 5 cycles. Appeared structurally sound after 5 cycles.

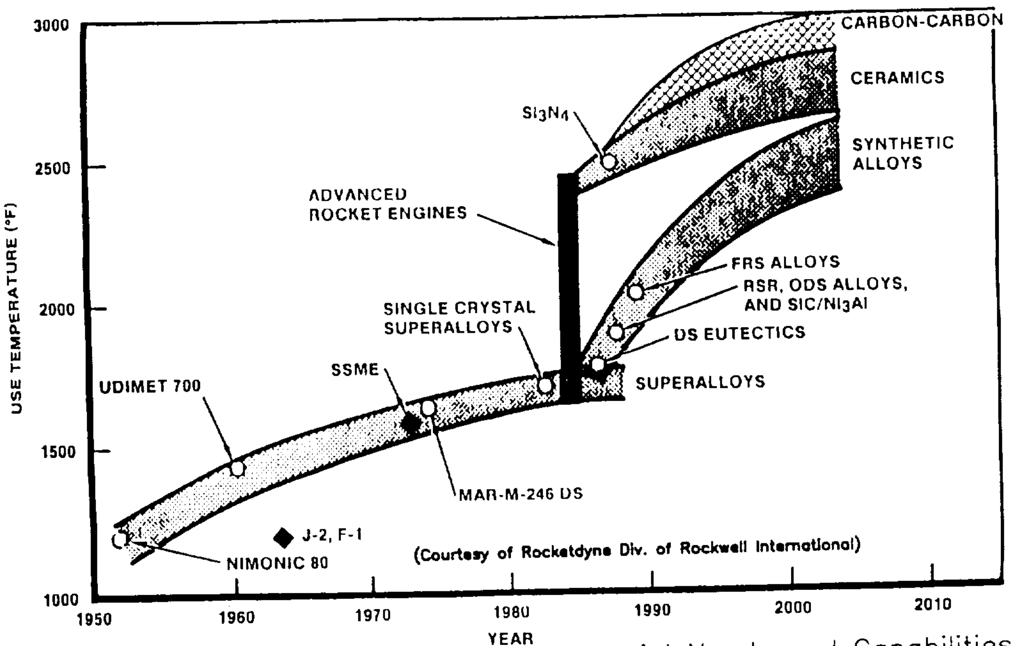

Figure 1. Rocket Engine Turbine Blade Material Needs and Capabilities

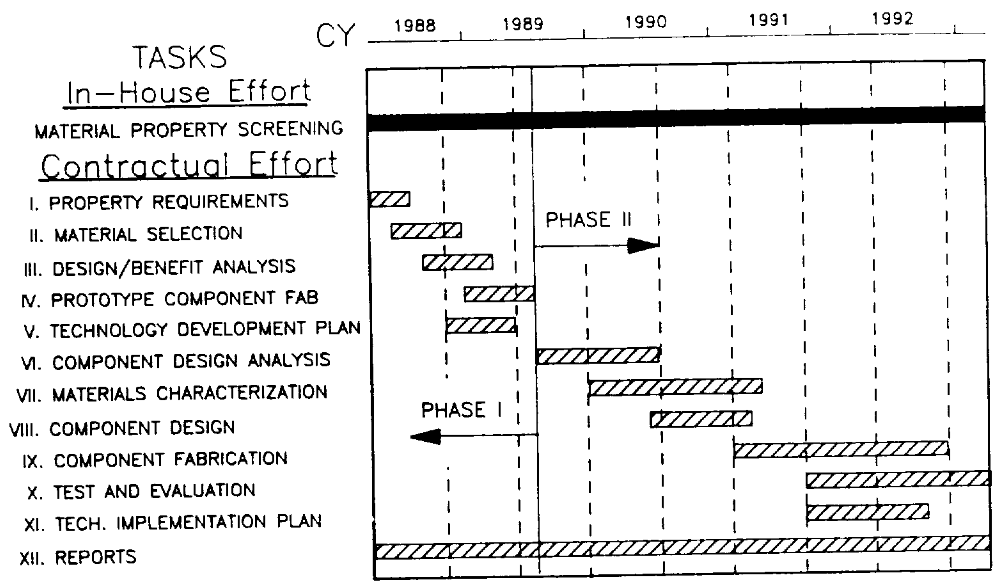

Figure 2. Program for Evaluating Advanced Fiber-Reinforced Ceramic Matrix Composites for Earth-to-Orbit Rocket Engine Turbines

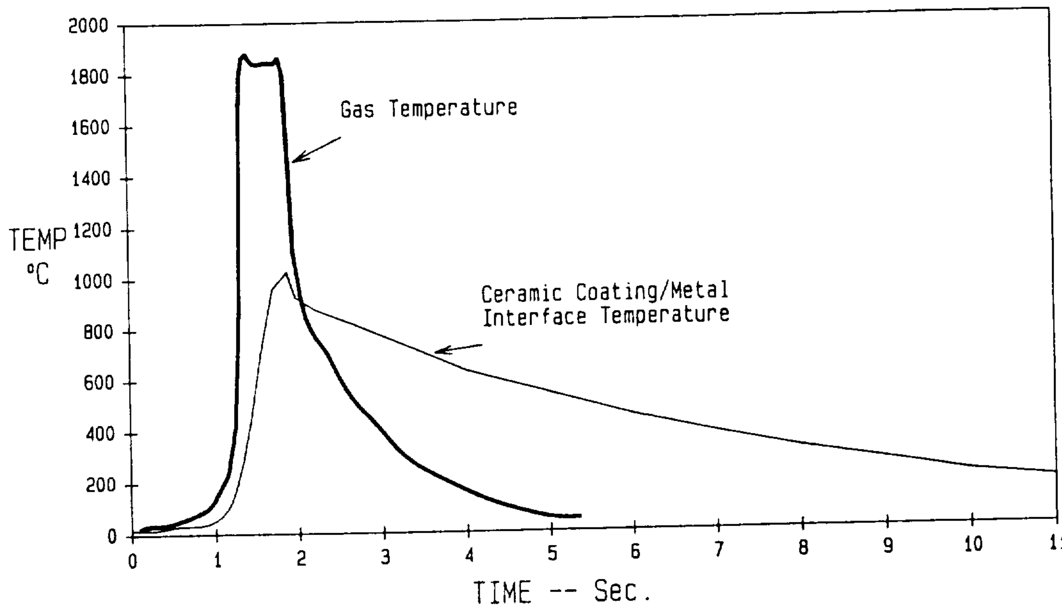

Fig. 3. Approximate thermal gradients generated for ceramic coated metallic substrate when operating at oxygen/hydrogen ration of 1:4.

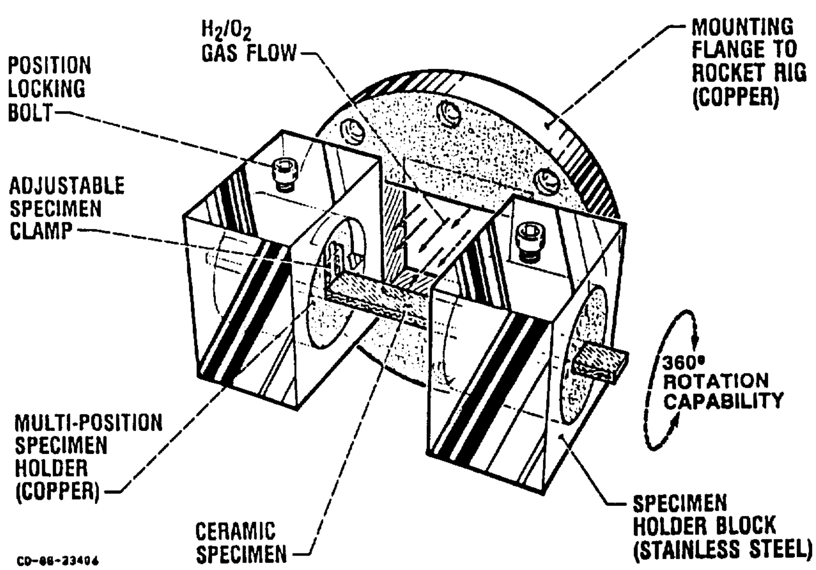

Figure 4. Thermal Shock Test Fixture

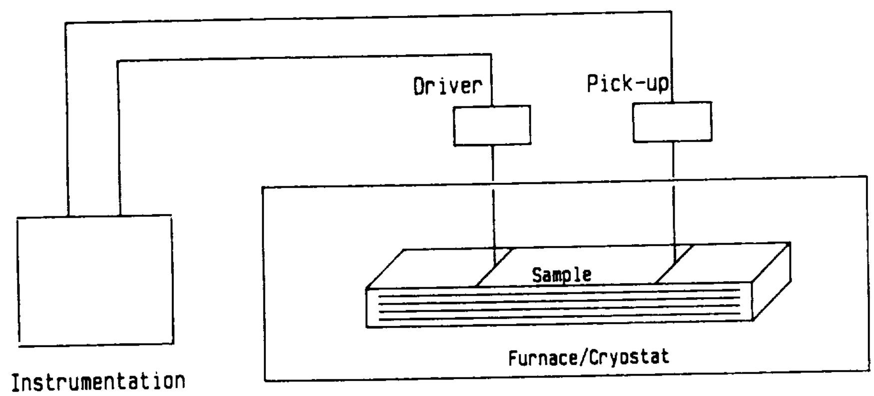

Fig. 5: Schematic of internal friction test fixture for measurement of thermal shock damage.

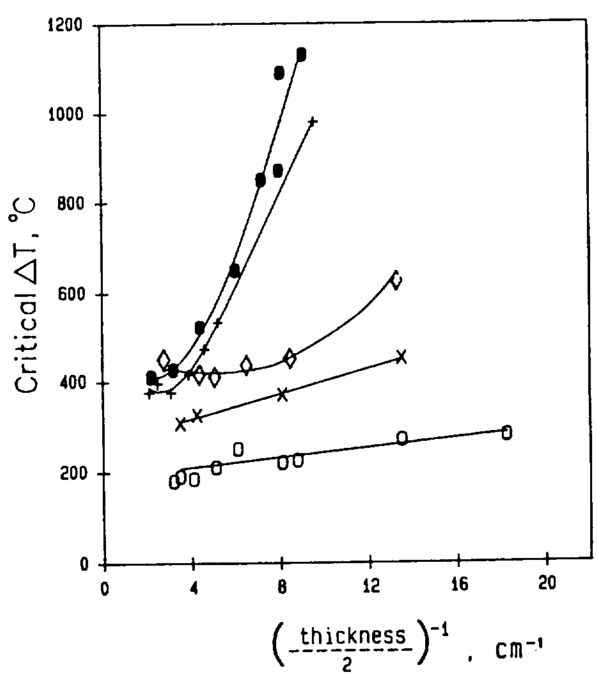

Fig. 6: Experimental results from Becher et al [7] for ΔT vs reciprocal specimen thickness for (O) alumina; (X) zirconia; (◊) glass ceramic; (+) borosilicate glass; and (●) silicon nitride.